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NOTES ON THE DETERMINATION OF MOLYBDENUM [1]

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A reliable mining company submitted to the Rare and Precious Metals Station of the Bureau of Mines a sample of oxidized lead ore containing a considerable quantity of vanadium and a small amount of molybdenum. It was stated that the sample was accurately quartered from similar samples sent to various assayors and chemists for analysis, that widely divergent returns for molybdenum were received, and that inaccurate sampling could not possibly account for the variable returns.

The amount of molybdenum reported by various chemists ranged from 3.14 per cent MoO_3 to 0.24 per cent MoO_3. As tested by the method described in this paper, the sample contained about 0.15 per cent MoO_3, a fair agreement with the lowest figure obtained elsewhere. With ores containing either vanadium or tungsten, the usual methods for determining molybdenum will yield high, erroneous results, unless special precautions are observed. From this and other similar instances it is evident that many assayers do not realize the necessity for precaution, even when they know interfering elements to be present.

In describing the quantitative determination of molybdenum most texts present a method of limited application and either omit or append the precautions necessary to guard against certain interfering elements. For ordinary custom work it is not safe to omit the procedure for separation of vanadium and tungsten from the molybdenum, unless the two elements are known to be absent. The following procedure has been selected as being the simplest reliable method for such occasional determinations as are encountered in custom analysis. For routine control work simpler volumetric methods may be preferable.

Heat 0.5 to 5 grams of the finely pulverized sample with nitric and then hydrochloric acid until decomposition is complete. Then add 7 c.c. of strong sulphuric acid and heat to fumes. Cool, add 50 c.c. of water, and heat to dissolve soluble sulphates. Cool, filter, and wash with cold dilute sulphuric acid, (1 to 10). The residue consisting of silica and lead sulphate should be tested for molybdenum unless perfectly white.

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Heat the filtrate, and add a slight excess of ammonia and 3 grams of sodium carbonate; the latter is necessary to prevent precipitation of molybdenum by large quantities of calcium. Boil 10 minutes; filter and wash twice with hot water. Wash the precipitate out of the funnel and digest it 15 minutes with more ammonia and soda. Again filter and wash well with hot dilute ammonia.

Add 5 grams of tartaric acid to the combined filtrates, warm, and saturate with hydrogen sulphide. If copper or other insoluble sulphides appear, they should be filtered off. The molybdenum remains in solution as thiomolybdate, which imparts a cherry red color to the solution. A slight excess of dilute sulphuric acid is next added, which precipitates molybdenum trisulphide, leaving vanadium and tungsten in solution. After heating a short time to coagulate, the precipitate is filtered and washed with hydrogen sulphide water containing a little sulphuric acid

To be certain that molybdenum is completely precipitated it is necessary to add nitric and sulphuric acids to the filtrate, evaporate to fumes, cool, add more nitric acid, and again fume to insure destruction of organic matter. Cool, add sufficient water to dissolve the salts, add ammonia, tartaric acid, and hydrogen sulphide, and precipitate the molybdenum as before. This precautionary procedure is seldom necessary in commercial work.

The molybdenum trisulphide is dissolved in hot dilute aqua regia, and the solution is neutralized with ammonia. Add 7 cc. of strong hydrochloric acid and 10 grams ammonium acetate. Dilute to 300 c.c., heat to boiling, and add slowly from a burette a solution of lead acetate until a test drop gives no color change with a drop of freshly-made tannic acid solution. Add a little more lead acetate, heat half an hour, and filter off the lead molybdate, washing with a 2 per cent solution of ammonium acetate.

The washed filter is placed in a fire clay annealing cup and ignited at red heat in a muffle. After cooling, the ignited precipitate can be brushed out of the annealing cup onto a balance pan and weighed directly. The weight of $PbMoO_4$ times 0.2615 gives the weight of Mo in the precipitate.

For a more complete discussion of this and other methods for estimating molybdenum the reader is referred to U. S. Bureau of Mines Bulletin 212, "Analytical Methods for Certain Metals." This bulletin may be found in the larger technical libraries, or may be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C., by remitting the price of 40 cents directly to that official.

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